

Attachment G

"Training Manual for Reviewing Laboratory Data Package
Completeness",

June 1994

TRAINING MANUAL FOR REVIEWING
LABORATORY DATA PACKAGE COMPLETENESS

June 1994

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Laboratory Data Package Completeness
Introduction

1.0 INTRODUCTION

When an analytical laboratory is requested/contracted by the Client to analyze field samples, the laboratory is required to provide adequate documentation supporting all current and future uses of the data. Potential uses of the data may include monitoring, modelling, risk assessment, site characterization, support of a remedy decision, and/or confirmation of treatment. Laboratory documentation and data may also be utilized in potential litigation as evidence.

Data packages produced by an analytical laboratory must contain all of the documents which were produced or used by the laboratory for that particular analysis. Although the specific documents required by the laboratory depends on the particular CLP RAS SOW or Client Request/Contract, in general, the laboratory data package must resemble as closely as possible the data packages required by the current CLP RAS SOWs for organics and inorganics. The tabulated summary forms provided in the SOWs must be utilized and modified appropriately, and qualifier flags such as those in the SOWs must be applied to the data as appropriate. For projects other than CLP RAS SOW projects, the data package must contain all modifications from the CLP RAS SOWs as specified in the Client Request/Contract.

This training manual provides procedures for reviewing laboratory data package completeness. Section 2.0 contains the specific laboratory documentation required in the data package, and Section 3.0 contains the specific information which must be provided on each document for both organic and inorganic analyses.

The required laboratory documentation and contents described in this manual closely resemble those required in the current CLP RAS SOWs for organics and inorganics. The exact format of the tabulated summary forms and specific documents required will depend on the particular analysis method and format requested/contracted by the Client. Sections 2.0 and 3.0 contain comprehensive laboratory data package requirements which can be modified according to the Client Request/Contract. A checklist of the required laboratory documentation and contents for organic and inorganic data are provided in Attachment V. Attachment VI contains the Region I CSF Completeness Evidence Audit Program

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document, dated July 3, 1991.

**Laboratory Data Package Completeness
Documentation**

2.0 LABORATORY DOCUMENTATION

The laboratory data package must adhere to the following general requirements:

- ! The data package must contain all original documents where possible
- ! The data package must be legible
- ! The data package must be clearly labeled and completed in accordance with Client instructions
- ! The data package must be arranged in increasing alphanumeric Client sample number order, or organized in a logical manner as specified by the Client Request/Contract
- ! The data package must be paginated consecutively in ascending order.

The laboratory data package documentation for both organic and inorganic analyses consists of the following comprehensive list:

1. Original sample data package including tabulated summary forms and raw data for field samples, standards, QC samples, and blanks (see below - sample data package)
2. A completed and signed Document Inventory Sheet used to record the inventory of the complete laboratory data package (see Attachments I and II for a comprehensive list of required documents for organics and inorganics, respectively)
3. All original shipping documents including, but not limited to, the following documents:
 - a. Client Chain-of-Custody Records/Traffic Reports
 - b. Airbills
 - c. Custody Seals
 - d. Sample tags (if present)
4. All original receiving documents including, but not limited to, the following documents:
 - a. Sample Log-In Sheet used to document the receipt and inspection of samples and containers
 - b. Other receiving forms or copies of receiving logbooks
 - c. Sample Delivery Group cover sheet identifying the samples received for the group of samples in the data package
5. All original laboratory records of sample transfer, preparation, and analysis including, but not limited to, the

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following documents:

- a. Original preparation and analysis forms and/or copies of preparation and analysis logbook pages
 - b. Internal sample and sample extract (organics) or sample digestate/distillate (inorganics) transfer chain-of-custody records
6. All other original project-specific documents in the possession of the laboratory including, but not limited to, the following documents:
- a. Telephone contact logs
 - b. Copies of personal logbook pages
 - c. All handwritten project-specific notes
 - d. All other project-specific documents not covered by the above.

The sample data package must include data for analysis of all samples in that Sample Delivery Group including the following:

1. Narrative
2. Tabulated summary forms for:
 - a. Field sample data (in increasing Client sample identification number)
 - b. Laboratory standards (in chronological order by instrument)
 - c. QC samples (in chronological order by type of QC sample)
 - d. Blanks (in chronological order by instrument)
3. Raw data for field samples, laboratory standards, QC samples, and blanks (in chronological order by instrument)
4. Laboratory logbook pages for preparation and analysis of field samples, standards, QC samples, and blanks
5. Chain-of-Custody Records
6. Other project-specific documents in the laboratory's possession.

In addition, for organic data each type of tabulated summary form must be grouped by fraction (volatile, semivolatile, pesticide/PCB). Depending on whether the data package contains organic or inorganic analytical data, the required tabulated forms and format for field samples, standards, QC samples, and blanks will vary. Section 3.0 describes the specific information

required for documentation of these analyses.

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3.0 LABORATORY DATA PACKAGE CONTENTS

The following sections list the information which must be provided on each laboratory document (listed below) in the sample data package for organic and inorganic analytical data:

1. Narrative
2. Tabulated summary forms
3. Raw data
4. Logbook pages
5. Chain-of-Custody Records.

Because of the differences in the required information between organic and inorganic analyses on the tabulated summary forms and raw data, these documents are discussed separately for organic and inorganic data (Sections 3.4.1 and 3.4.2, respectively). The narrative, logbook pages, and Chain-of-Custody Record documentation for both organic and inorganic analyses require similar information and are discussed together below (Sections 3.1, 3.2, and 3.3). The specific requirements resemble those of the current CLP RAS SOWs and must be modified as appropriate to the Client Request/Contract.

3.1 Narrative

The narrative must describe the analytical methods and exact procedures performed by the laboratory as well as any deviations from the methods. The laboratory must document in detail all problems encountered with quality control, samples, shipment, and all analytical problems encountered in processing the samples. Problem resolution must be documented as well as any other factors which may affect the validity of the data. The laboratory must also discuss all unusual occurrences encountered during the analysis of the sample set. The laboratory must explain all data flags if not specified in the analytical method.

Additionally, for CLP RAS specific organic data the laboratory must list the pH of each water sample submitted for volatiles analysis. The laboratory must also list all instances of manual integrations performed by analysts. For CLP RAS specific inorganic data, the laboratory must indicate whether ICP interelement corrections and background corrections were applied.

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The laboratory must provide examples of calculations of both a detected positive result and a detection/quantitation limit reported for each type of sample analysis. All equations, sample volumes, sample weights, dilution factors, percent solids/percent moisture, and other information required to reproduce the laboratory results must be indicated.

The narrative must also include the following additional items of information:

- ! Laboratory name
- ! Client Request/Contract project number
- ! CLP RAS or other Client sample identification numbers cross-referenced to the laboratory sample identification numbers.

In addition, the narrative must be signed and dated by the laboratory manager or designee.

Communication Logs

All telephone communications between the laboratory and sampling personnel or other parties outside the laboratory, which took place to resolve sampling discrepancies or analytical problems must be documented in detail on telephone communication logs. Those telephone logs must explicitly detail the problems discussed, the resolution agreed upon, the names and affiliations of the communicating parties, and the date the communication took place. All telephone logs must be appended to the narrative.

3.2 Laboratory Logbook Pages

The data package must contain the following laboratory logbook pages. Where possible, the originals must be submitted.

- ! Standards preparation logs
- ! QC sample preparation logs
- ! Sample preparation/extraction/digestion logs
- ! Sample analysis run logs
- ! Personal logs
- ! Hand-written project-specific notes.

The logbook pages must contain the following information where applicable:

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! Laboratory name
! Client sample identification number
! Laboratory sample identification number
! Dates of preparation and analysis, and initials of preparer/analyst
! Source of standards and QC samples
! Weights and volumes of samples and standards
! Initial and final volumes of sample prepared/purged/extracted/digested
! Percent moisture/percent solids
! Injection/analysis volumes
! Date and time of sample injection
! Dilution factors.

3.3 Chain-of-Custody Records

Documentation must be provided of the traceability of the Client samples from the time the samples are released to the laboratory and while in the laboratory's possession. Two types of custody records must be provided in the data package: records of external sample transfer and custody from parties outside the laboratory, and records of internal sample transfer and custody within the laboratory.

On the external Chain-of-Custody Record, usually initiated by the Client/sampler, the laboratory is responsible for providing the following information:

- ! Date of sample receipt
- ! Signature of receiving personnel
- ! Condition of shipping containers and sample bottles upon receipt
- ! Condition of custody seals
- ! Presence/absence of airbills, custody seals, Client custody records, traffic reports, sample tags
- ! Problems or discrepancies with samples received or the documentation on the Chain-of-Custody Record.

The Chain-of-Custody Record also contains other vital information (e.g. sampling date/time, etc.) but documentation of this information is the responsibility of the Client/sampler.

The laboratory's internal Chain-of-Custody Records must contain the following information:

- ! Laboratory name
- ! Client sample identification number
- ! Laboratory sample identification number
- ! Date of sample transfer and receipt
- ! Signature of personnel transferring/receiving the sample
- ! Purpose of transfer/receipt, location of sample transferred.

3.4 Tabulated Summary Forms and Raw Data

The exact format of the tabulated summary form for each field sample, QC sample, standard, and blank will depend on the particular analysis method requested/contracted by the Client.

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The laboratory must provide certain information on all the tabulated summary forms and raw data: laboratory name, project/contract number, concentration units wherever numerical values are reported, and indication of wet or dry weight for solid matrices. The following sections list the specific requirements for organic data and inorganic data.

3.4.1 ORGANIC FORMS AND RAW DATA

Information required on tabulated summary forms are presented in the following sections for the organic data listed below:

1. Field sample results
2. Surrogate results (system monitoring compound results)
3. Matrix spike/matrix spike duplicate results
4. Method/laboratory blank results
5. Tuning results (GC/MS instrument performance check)
6. Initial calibration results (GC/MS)
7. Initial calibration results (GC)
8. Continuing calibration results (GC/MS)
9. Continuing calibration results (GC)
10. Internal standard results (GC/MS)
11. GC analytical sequence
12. Pesticide cleanup results
13. Pesticide/PCB identification summary
14. Method detection limit study results

Tabulated summary forms for organic data required by the CLP RAS SOWs are provided in Attachment III as examples.

Field Sample Results (Form 1)

Comprehensive tabulated summary forms must be prepared for each field sample analyzed by the laboratory. At a minimum, the tabulated summary forms must contain the following information:

- ! Client sample identification (ID) number
- ! Laboratory sample ID number
- ! Target compound names
- ! Tabulated analytical results for identification (numerical quantitation limits) and quantitation (positive hits) with concentration units
- ! Any laboratory qualifier flags - laboratory qualifier flags for each target analyte must be tabulated on a separate form (definitions must be provided for each laboratory qualifier flags).

For each field sample, the tabulated summary forms must also contain the following information as appropriate to the analysis method:

! Laboratory file ID
! Sample matrix type
! Level of analysis (low, medium)
! Percent moisture or percent solids
! GC column
! Sample weights and/or sample volumes prepared/purged/
extracted/analyzed
! Initial and final extract and extract clean-up volumes,
injection volume
! Clean-ups performed
! Dilution factor
! Measured pH
! Dates of sample receipt, extraction, and analysis.

Surrogate Results (System Monitoring Compound Results) (Form 2)

Surrogate recovery data help to evaluate the efficiency of the sample preparation and analysis procedures and analytical system. The tabulated surrogate results summary form must contain the following information:

! Sample matrix
! Level of analysis (low, med)
! GC column
! Client sample ID numbers
! Surrogate compounds added
! Percent recoveries of surrogates
! QC limits for all surrogate standards in field samples, QC samples, and blanks
! All outliers flagged
! Total number of surrogates outside QC limits
! Indication of surrogates diluted out.

Matrix Spike/Matrix Spike Duplicate Results (Form 3)

Matrix spike and matrix spike duplicate samples are analyzed to evaluate the effects of the sample matrix on the methods used for analysis. The tabulated MS/MSD results summary form must contain the following information:

! Sample matrix
! Level of analysis (low, med)
! Client sample ID number

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- ! Matrix spike compounds added
 - ! True concentrations of the spikes added
 - ! Concentrations of the spike compounds observed in the spiked sample
 - ! Sample concentration of each spike compound detected in the original unspiked sample for the MS and MSD
 - ! Percent recoveries of the spiked compounds in the MS/MSD samples
 - ! Relative percent differences of the spiked compounds between the MS and MSD samples
 - ! QC limits for all spike compounds - percent recovery and relative percent difference
 - ! All outliers flagged.

In addition to the above, the results for all target compounds in the MS and MSD samples must be tabulated on the summary forms used to tabulate the field sample results.

Method/Laboratory Blank Results (Form 4)

The laboratory must provide blank information to determine the levels of contamination associated with the processing and analysis of the samples for method blanks and, depending on the analysis method, laboratory (instrument) blanks. The tabulated method/laboratory blanks results form must contain the following information:

- ! GC column, instrument ID
- ! Date and time of analysis for the blank itself
- ! Date of extraction
- ! Matrix with which the blank is associated
- ! Level of analysis (low, med)
- ! Laboratory sample ID number
- ! List of Client field sample ID numbers and MS/MSD samples associated with each blank (separate forms are used for each blank)
- ! Laboratory file IDs of the samples and associated blank
- ! Dates/times of analysis for field samples and MS/MSD samples which are associated with each blank.

In addition, results for each method and laboratory instrument blank must be included on the tabulated summary forms that are used for field sample results (Form 1).

Tuning Results (GC/MS Instrument Performance Check) (Form 5)

For GC/MS analyses, the laboratory must perform instrument performance checks to assure correct mass calibration, mass resolution, and mass transmission. The tabulated GC/MS tuning results summary form must contain the following information:

- ! Instrument ID, laboratory file ID
- ! Date and time of injection for each tune compound analysis (each tune on a separate form)
- ! Tune compound name
- ! Mass-to-charge ratio (m/e) for each ion
- ! Ion abundance criteria

! Percent relative abundances.

The form must also contain the following tabulated information associated with each tune and in chronological order:

! Client sample ID numbers associated with that tune
! Laboratory sample ID numbers
! Laboratory file IDs
! Date and time of analysis for all field samples, MS/MSD samples, blanks, and standards associated with that tune
! All outliers flagged.

Initial Calibration Results (GC/MS) (Forms 6A-6C)

Prior to any analysis, the laboratory must initially calibrate the GC/MS system to determine the linearity of the response. The tabulated GC/MS initial calibration results summary form must contain the following information:

! Instrument ID, laboratory file IDs
! Purge method
! Dates and times of standard analyses for that initial calibration
! Target compound names
! Concentrations of the calibration standards
! Relative response factors for each target and surrogate compound at each standard concentration
! Mean relative response factors for each target and surrogate compound
! Percent relative standard deviations for each target and surrogate compound
! QC limits for each initial calibration (each initial calibration on a separate form) - minimum RRF, maximum % RSD values
! All outliers flagged.

Initial Calibration Results (GC) (Forms 6D-6G)

Because the identification of compounds using GC is based primarily on retention time data or pattern recognition, the retention times and retention time windows are crucial to the provision of valid data. Generally, the tabulated initial calibration results summary forms for GC systems consist of

retention time and calibration factor information. The data for pesticides, generally multi-point calibrations, and PCBs, generally single-point calibrations, are usually provided on two separate forms.

The following retention time information must be documented on the initial calibration results summary form:

- ! Instrument ID, GC column
- ! Dates of analysis
- ! Concentration of the calibration standards
- ! Target compound and surrogate compound names
- ! Retention times for each target and surrogate compound at each standard concentration
- ! Mean retention times for each target and surrogate compound (if multi-point calibration)
- ! Retention time windows for each target and surrogate compound (QC limits).

The following calibration factor information must be documented as well:

- ! Instrument ID, GC column
- ! Dates of analysis
- ! Concentrations of the calibration standards
- ! Target compound and surrogate compound names
- ! Calibration factors for each target and surrogate compound at each standard concentration
- ! Mean calibration factor (for multi-point calibration) for each target and surrogate compound
- ! Percent Relative Standard Deviation for each target and surrogate compound
- ! QC limits - % RSD
- ! All outliers flagged.

Resolution between compounds is documented with the following information:

- ! Instrument ID, GC column
- ! Dates and times of analysis
- ! Laboratory sample ID
- ! Names of compounds for which resolution is measured
- ! Retention times for each of those compounds

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- ! Percent resolution between each pair of compounds
 - ! QC limits - % resolution
 - ! All outliers flagged.

Continuing Calibration Results (GC/MS) (Form 7A-7C)

The continuing calibration standards are analyzed to verify the accuracy of the initial calibration. The tabulated continuing calibration results form must contain the following information:

- ! Instrument ID, laboratory file ID
- ! Purge method
- ! Date and time of continuing calibration analysis
- ! Date and time of initial calibration analysis associated with that continuing calibration
- ! Target compound and surrogate compound names
- ! Mean relative response factors from initial calibration for each target and surrogate compound
- ! Relative response factors from continuing calibration for each target and surrogate compound (each continuing calibration on a separate form)
- ! Percent differences for each compound
- ! QC limits for each target and surrogate compound (each continuing calibration on a separate form) - minimum RRF, maximum % D
- ! Concentrations of the continuing calibration standards
- ! All outliers flagged.

Continuing Calibration Results (GC) (Forms 7D, 7E)

The tabulated continuing calibration results form must contain the following information:

- ! Instrument ID, GC column
- ! Laboratory sample ID
- ! Dates and times of continuing calibration standards analysis
- ! Date of associated initial calibration analysis
- ! Target compound and surrogate compound names
- ! Retention time for each target and surrogate compound
- ! Calculated amount of standard
- ! Nominal amount of standard
- ! Relative Percent Difference for each compound
- ! QC control limits - RPD

-
- ! Percent breakdowns for compounds used to measure extent of breakdown (endrin and 4,4'-DDT) and combined breakdown
 - ! QC limits - percent breakdown.

Internal Standard Results (GC/MS) (Forms 8A-8C)

Internal standard responses in all calibration standards, field samples, QC samples, and blanks are crucial to the provision of reliable analytical results because the internal standards are used to quantitate the compounds. The tabulated internal standard results summary forms must contain the following information.

- ! Instrument ID, laboratory file ID
- ! GC column, purge method
- ! Date and time of continuing calibration standard analysis
- ! Client sample identification numbers
- ! Internal standard compound names
- ! Retention times and area counts of the quantitation for each internal standard compound in the continuing calibration standard, field samples, MS/MSD samples, and blanks associated with that continuing calibration (separate form for each continuing or initial calibration)
- ! QC limits - area counts and retention times
- ! All outliers flagged.

GC Analytical Sequence (Form 8D)

The Client Request/Contract may require standards and samples to be analyzed according to a special sequence. In this case, the following information must be provided:

- ! Instrument ID, GC column
- ! Initial calibration dates
- ! List of Client sample ID numbers in that analytical sequence (in chronological order) for all standards, field samples, QC samples, and blanks
- ! Laboratory sample ID numbers
- ! Dates and times of analyses
- ! Mean surrogate retention times - from initial calibration
- ! Retention times of the surrogate compounds

! QC limits of the surrogates - retention times
! All outliers flagged.

Pesticide Cleanup Results (Form 9)

Tabulated summary forms may be required when cleanup procedures are employed during the preparation of pesticide extracts for analysis. The following information are documented for reporting the results of the check of the Florisil cartridges used to process samples and extracts, and to summarize the results of the calibration of the Gel Permeation Chromatography (GPC) used to process soil sample extracts for Pesticide/PCB analyses.

Florisil Cartridge Check Results (Form 9A):

! GC column
! Florisil cartridge lot number (each lot on a separate form)
! Date of check solution analysis
! Names of compounds in the Florisil cartridge check solution
! Amount of spike in the check solution
! Amount of spike recovered in the check solution
! Percent recoveries
! QC limits - percent recoveries
! All outliers flagged
! Client sample ID numbers associated with that Florisil cartridge
! Laboratory sample ID numbers associated with that Florisil cartridge
! Dates of sample analysis.

GPC Calibration Results (Form 9B):

! GPC column
! Calibration date of GPC column
! GC columns
! Name of spike compounds added to GPC column
! Amount of spike added
! Amount of spike recovered
! Percent recoveries
! QC limits - % recoveries
! All outliers flagged
! Client sample ID numbers associated with the GPC column calibration

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- ! Laboratory sample ID numbers associated with the GPC column calibration
 - ! Dates of sample analyses.

Pesticide/PCB Identification Summary (Form 10)

This form summarizes the quantitations of all target Pesticide/PCB compounds detected in each field sample, QC sample, and blanks. If no compounds are detected in a given sample, this form is not required.

- ! Instrument ID, GC columns
- ! Dates of analysis
- ! Client sample ID number (on a separate form for each sample)
- ! Laboratory sample ID number
- ! Target compound name detected
- ! Retention time of compound on each column
- ! Retention time windows
- ! Concentration (mean concentration for multicomponent compounds)
- ! Percent difference.

Method Detection Limit Study Results

The tabulated MDL study results must contain the following information:

- ! Target compound names
- ! Concentrations of spikes added
- ! Concentration detected for each MDL spike
- ! Standard deviation and calculated MDL for each target compound.

The exact procedure utilized to generate the MDLs must be documented in detail in the narrative. The equation and associated constant values utilized to calculate the MDL for each analysis must be documented. The column, instrument ID, trap composition, and operating conditions must be clearly documented in the raw data.

Raw Data

The laboratory data package must contain raw data for all field samples, standards, QC samples, matrix spike and matrix spike duplicate samples, and blanks. The exact format and content of the raw data will depend on the particular analysis method requested/contracted. However, all instrument printouts, strip chart recordings, chromatograms, quantitation reports, mass spectra, and other types of raw data generated by the laboratory for a particular project must be provided in the data package.

Typical raw data for organic GC/MS analyses includes, but is not limited to the following:

- ! Reconstructed total ion chromatogram for each sample or sample extract, standards, QC samples, and blanks
- ! Instrument quantitation reports containing the following information: laboratory sample identification number, Client sample identification number, date and time of analysis, retention time and/or scan number of quantitation ion with measured area, analyte concentration, copy of area table from data system, GC/MS instrument ID, laboratory file ID, column, trap composition, and operating conditions
- ! Raw and enhanced mass spectra for all positive target compound results in field samples; daily continuing calibration standard reference spectra for all positive field sample results
- ! Mass spectra and three library searched best-match mass spectra for all tentatively identified compounds reported
- ! Instrument normalized mass listing and the mass spectrum for each tune.

Typical raw data for organic GC analyses includes, but is not limited to the following:

- ! Chromatograms for field samples, calibration standards, QC samples, and blanks containing the following information: Client sample identification number, laboratory sample identification number, volume injected, date and time of injection, GC column identification, GC instrument identification, laboratory file ID, operating conditions, positively identified compounds must be labeled with the compound names either directly from the peak or on a printout of the retention times
- ! Chromatograms for both GC columns

- ! GC integration report or data system printout
- ! Manual worksheets.

3.4.2 INORGANIC FORMS AND RAW DATA

Tabulated summary form requirements are presented in the following sections for the inorganic data listed below:

1. Field sample results
2. Initial and continuing calibration verification results
3. Contract required detection limit standard results
4. Blank results
5. ICP interference check sample results
6. Matrix spike and post-digestion spike sample results
7. Duplicate sample results
8. Laboratory control sample results
9. Method of standard additions results
10. ICP serial dilution results
11. Instrument detection limits
12. ICP interelement correction factors
13. ICP linear ranges
14. Preparation log
15. Analysis run log and Furnace AA QC results.

Tabulated data reporting forms for inorganic data required by the CLP RAS SOWs are provided in Attachment IV as examples.

Field Sample Results (Form 1)

Comprehensive tabulated summary forms must be prepared for each field sample analyzed by the laboratory. At a minimum, the tabulated summary forms must contain the following information as appropriate to the analysis method:

- ! Sample matrix type
- ! Level of analysis (low, medium)
- ! Percent moisture or percent solids
- ! Date of sample receipt
- ! Client sample identification number
- ! Laboratory sample identification number
- ! Target analyte names
- ! Tabulated analytical results for identification (numerical detection/quantitation limits) and quantitation (positive hits) with concentration units
- ! Any laboratory qualifier flags - laboratory qualifier flags for each target analyte must be tabulated on a separate form

(definitions must be provided for each laboratory qualifier flags)

- ! Concentration qualifier - indication of results less than the contract required detection limits
- ! Analytical method used for each analyte (usually indicated with a symbol).

Information regarding sample weights, volumes, dilution factors, and dates of digestion and analysis which are provided on the field sample results summary forms for organic data are not typically provided on those forms for inorganic data. Instead, this information is provided on other tabulated forms which follow.

Initial and Continuing Calibration Verification Results (Form 2A)

Initial and continuing calibration verification standards are analyzed to verify and ensure the accuracy of the initial and continuing calibrations. The tabulated initial and continuing calibration verification results summary form must contain the following information:

- ! Sources of the initial and continuing calibration verification standards
- ! Target analyte names
- ! True values of the calibration verification standards
- ! Concentrations found for the calibration verification standards
- ! Percent recoveries
- ! QC limits - percent recoveries
- ! Analytical method used for each analyte (usually indicated with a symbol).

The order of reporting the initial and continuing calibration verification standards for each analyte must follow the order in which the standards were analyzed.

Contract Required Detection Limit Standard Results (Form 2B)

Contract required detection limit (CRDL) standards are analyzed to verify the linearity of the instrument near the contract required detection limit. The tabulated CRDL results form must contain the following information:

! Source of the CRDL standards
! Target analytes
! True values of the CRDL standard for each analyte
! Concentrations found for each analyte
! Percent recoveries for each analyte
! QC limits (if known).

The order of reporting the CRDL standard results for each analyte must follow the order in which they were analyzed.

Blank Results (Form 3)

The tabulated blank results summary form must, in general, contain information for two types of blanks: initial and continuing calibration blanks, or instrument blanks, and preparation blanks. The tabulated blank results summary form must contain the following information:

! Matrix for which the preparation blank is associated
! Concentration units of each blank type
! Target analyte names
! Initial and continuing calibration blank results
! Preparation blank results
! Concentration qualifiers
! Analytical method used for each analyte (usually indicated with a symbol).

The order of reporting the initial and continuing calibration blanks and preparation blanks for each analyte must follow the order in which they were analyzed.

ICP Interference Check Sample Results (Form 4)

ICP interference check samples are analyzed to verify interelement and background correction factors by analyzing target analytes in the presence of interferences. The tabulated ICP interference check sample results forms must contain the following information:

! ICP instrument ID number
! Source of the ICS solutions
! Target ICP analytes
! True values of each target ICP analyte in the solution

-
- containing interferents only
 - ! True values of each target ICP analyte in the solution containing interferents and analytes
 - ! Concentrations of target ICP analytes detected in the solution containing interferents only
 - ! Concentrations of target ICP analytes detected in the solution containing interferents and analytes
 - ! Percent recoveries.

The order of reporting the interference check sample results for each analyte must follow the order in which they were analyzed.

Matrix Spike and Post-Digestion Spike Sample Results (Form 5A, 5B)

The matrix spike sample analysis provides information about the effect of the sample matrix on the digestion and measurement methodology. The post-digestion spike recovery is based on the addition of a known quantity of analyte to an aliquot of digested sample. The tabulated matrix spike and post-digestion spike sample results (Forms VA and VB, respectively) must contain the following results:

- ! Sample matrix
- ! Level of analysis (low, medium)
- ! Percent solids of the sample
- ! Client sample identification number
- ! Target analyte names
- ! Concentrations of the spikes added to the sample
- ! Concentrations found in the spiked sample
- ! Concentration found in the unspiked sample
- ! Percent recoveries
- ! QC limits - percent recovery
- ! All outliers flagged
- ! Concentration qualifiers
- ! Analytical method used for each analyte (usually indicated with a symbol).

Duplicate Sample Results (Form 6)

Duplicate sample analysis provide information about the laboratory precision. The tabulated duplicate sample results form must contain the following information:

- ! Sample matrix
- ! Level of analysis (low, medium)
- ! Percent solids of the original sample and duplicate sample
- ! Client sample identification number
- ! Target analyte names
- ! Concentration of the original sample result
- ! Concentration of duplicate sample result
- ! Relative percent difference
- ! QC limits
- ! All outliers flagged
- ! Concentration qualifiers
- ! Analytical method used for each analyte (usually indicated with a symbol).

Laboratory Control Sample Results (Form 7)

Laboratory control sample results provide information about the efficiency of the digestion method and accuracy of the results. The tabulated laboratory control sample results form must contain the following information:

- ! Source of the laboratory control sample
- ! Matrix of the LCS
- ! Target analyte names
- ! True concentrations
- ! Concentrations found
- ! Percent recoveries
- ! QC limits
- ! Concentration qualifiers.

Because a laboratory control sample should be digested for each matrix and digestion batch, additional forms must be present as appropriate if more than one LCS for a matrix was analyzed.

Method of Standard Additions Results (Form 8)

The method of standard additions analysis may be performed by the laboratory to quantitate the analyte in the sample when matrix interferences are present. The tabulated method of standard additions results form must contain the following information:

- ! Client sample identification number
- ! Concentrations of each MSA spike added

- ! Absorbance detected in each MSA spike as well as the sample itself
- ! Final concentration
- ! Correlation coefficient
- ! All outliers flagged.

Results for different samples for each analyte must be reported sequentially.

ICP Serial Dilution Results (Form 9)

ICP serial dilution analyses provide information as to the extent of the matrix effects in the sample. The tabulated ICP serial dilution results form must contain the following information:

- ! Sample matrix
- ! Level of analysis (low, medium)
- ! ICP instrument ID
- ! Client sample identification number
- ! Target analyte names
- ! Concentrations of the undiluted sample result
- ! Concentrations of the diluted sample result
- ! Percent difference
- ! QC limits - percent difference
- ! All outliers flagged
- ! Concentration qualifiers
- ! Analytical method used for each analyte (usually indicated with a symbol).

Instrument Detection Limits (Form 10)

The tabulated instrument detection limit results form must contain the following information:

- ! Instrument ID numbers used for the IDL determination
- ! Date the IDLs were determined
- ! Wavelength and background used for each analyte
- ! Target analyte names
- ! Type of background correction used (where applicable)
- ! Instrument detection limits
- ! Contract required detection limits
- ! Concentration units
- ! Analytical method used for each analyte (usually indicated

with a symbol.

ICP Interelement Correction Factors (Form 11)

This form must document for each ICP instrument used for analysis, the interelement correction factors applied by the laboratory to obtain the reported data. The tabulated ICP interelement correction factors form must contain the following information:

- ! ICP instrument ID number
- ! Wavelength for each analyte used for the determination
- ! Date of interelement correction factor determination
- ! Target ICP analyte names
- ! Interfering analytes with which the interelement correction factors were determined
- ! Interelement correction factors for each analyte.

ICP Linear Ranges (Form 12)

A linear range verification check standard must be analyzed and reported for each target analyte as this concentration is the upper limit of the ICP linear range beyond which results should not be reported without dilution of the sample. The tabulated ICP linear ranges must contain the following information:

- ! ICP instrument ID number
- ! Date of the linear range determination
- ! Integration time for each analyte
- ! Target ICP analytes
- ! Concentration of the upper limit of the linear range for each analyte.

Preparation Log (Form 13)

This form provides sample preparation information which documented in the laboratory logbook pages. The following information is required:

- ! Analytical method (each analytical method on a separate form)
- ! Client sample ID number of all field samples, QC samples, standards, and blanks digested/distilled

- ! Sample preparation date
- ! Sample weight
- ! Sample volume.

Analysis Run Log and Furnace AA QC Results (Form 14)

This form provides information as to the analytical sequence of each analyte and any dilution factors applied. The tabulated analysis run log summary form must provide the following information.

- ! Instrument ID
- ! Analytical method
- ! Start and end dates of the analytical sequence
- ! Client sample ID numbers in chronological order
- ! Dilution factors
- ! Time of analysis for each analytical sample and standard
- ! Analytes associated with the run sequence.

Furnace AA QC analysis results are also typically provided on this form. Because of the nature of the furnace AA technique, the Client Request/Contract may require special QC sample analyses for quantitation of field samples. The QC samples which may be required are the following: duplicate injections of each analytical and field sample, and post-digestion spikes of each field sample. For furnace AA analyses, the following additional information are required on the Analysis Run Log Form:

- ! Percent relative standard deviation of duplicate injections (outliers usually indicated with flags on the tabulated field sample results summary form, Form 1)
- ! Percent recoveries of post-digestion spikes

The above information must be reported on separate forms for each furnace AA analyte.

Raw Data

The laboratory data package must contain raw data for all field samples, standards, QC samples, matrix spike and duplicate samples, and blanks. The exact format and content of the raw data will depend on the particular analysis method requested/contracted. However, for each reported value for a particular

project, the laboratory must include all raw data used to obtain that reported value.

Typical raw data for inorganic analyses include, but is not limited to the following:

- ! Instrument printouts, strip chart recordings, etc., for all field samples, QC samples, standards, and blanks containing the following information: laboratory sample identification number, Client sample ID number, date and time of analysis, absorbance/emission values, analyte concentration, instrument ID, lab file ID, and instrument operating conditions.
- ! Standard curve raw data, plotted standard curves, linear regression equations, and correlation coefficients.

4.0 ACRONYMS

AAS	Atomic Absorption Spectrometry
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
GPC	Gel Permeation Chromatography
ICP	Inductively Coupled Plasma
ICS	Interference Check Sample
ICV	Initial Calibration Verification
ID	Identification
IDL	Instrument Detection Limit
LCS	Laboratory Control Sample
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSA	Method of Standard Additions
PB	Preparation Blank
QC	Quality Control
RAS	Routine Analytical Services

RPD Relative Percent Difference

RRF Relative Response Factor

RSD Relative Standard Deviation

SOW Statement of Work

ATTACHMENT I

ORGANIC DOCUMENT INVENTORY CHECKLIST

For hardcopy of Attachment I contact:

Steve Stodola, U.S. EPA Region I

TEL: 781-860-4634

EMAIL: stodola.steve@epamail.epa.gov

ATTACHMENT II

INORGANIC DOCUMENT INVENTORY CHECKLIST

For hardcopy of Attachment II contact:

Steve Stodola, U.S. EPA Region I

TEL: 781-860-4634

EMAIL: stodola.steve@epamail.epa.gov

ATTACHMENT III

EXAMPLE ORGANIC TABULATED SUMMARY FORMS

For hardcopy of Attachment III contact:

Steve Stodola, U.S. EPA Region I

TEL: 781-860-4634

EMAIL: stodola.steve@epamail.epa.gov

ATTACHMENT IV

EXAMPLE INORGANIC TABULATED SUMMARY FORMS

For hardcopy of Attachment IV contact:

Steve Stodola, U.S. EPA Region I

TEL: 781-860-4634

EMAIL: stodola.steve@epamail.epa.gov

ATTACHMENT V

LABORATORY DOCUMENTATION AND CONTENTS CHECKLIST
FOR ORGANIC AND INORGANIC DATA

LABORATORY DATA PACKAGE COMPLETENESS

DOCUMENTATION CHECKLIST

COMPLETE LABORATORY DATA PACKAGE DOCUMENTATION	
1.	Original <u>sample data package</u> including tabulated summary forms and raw data for field samples, standards, QC samples, and blanks (see below - sample data package)
2.	A completed and signed Document Inventory Sheet used to record the inventory of the complete laboratory data package
3.	All original shipping documents including, but not limited to, the following documents: <ul style="list-style-type: none"> a. Client Chain-of-Custody Records/Traffic Reports b. Airbills c. Custody Seals d. Sample tags (if present)
4.	All original receiving documents including, but not limited to, the following documents: <ul style="list-style-type: none"> a. Sample Log-In Sheet used to document the receipt and inspection of samples and containers b. Other receiving forms or copies of receiving logbooks c. Sample Delivery Group cover sheet identifying the samples received for the group of samples in the data package
5.	All original laboratory records of sample transfer, preparation, and analysis including, but not limited to, the following documents: <ul style="list-style-type: none"> a. Original preparation and analysis forms and/or copies of preparation and analysis logbook pages b. Internal sample and sample extract (organics) or sample digestate/distillate (inorganics) transfer chain-of-custody records
6.	All other original project-specific documents in the possession of the laboratory including, but not limited to, the following documents: <ul style="list-style-type: none"> a. Telephone contact logs b. Copies of personal logbook pages c. All handwritten project-specific notes d. All other project-specific documents not covered by the above
SAMPLE DATA PACKAGE DOCUMENTATION	
1.	Narrative
2.	Tabulated summary forms for <ul style="list-style-type: none"> ! Field sample data (in increasing Client sample identification number) ! Laboratory standards (in chronological order by instrument) ! QC samples (in chronological order by type of QC sample) ! Blanks (in chronological order by instrument)
3.	Raw data for field samples, laboratory standards, QC samples, and blanks (in chronological order by instrument)
4.	Laboratory logbook pages for preparation and analysis of field samples, standards, QC samples, and blanks
5.	Chain-of-Custody Records
6.	Other project-specific documents in the laboratory's possession
<p>For organic data each type of tabulated summary form must be grouped by fraction (volatile, semivolatile, pesticide/PCB). Depending on whether the data package contains organic or inorganic analytical data, the required tabulated forms and format for field samples, standards, QC samples, and blanks will vary.</p>	

LABORATORY DATA PACKAGE COMPLETENESS

DOCUMENTATION CHECKLIST

ORGANIC TABULATED SUMMARY FORMS	
1.	Field sample results
2.	Surrogate results (system monitoring compound results)
3.	Matrix spike/matrix spike duplicate results
4.	Method/laboratory blank results
5.	Tuning results (GC/MS instrument performance check)
6.	Initial calibration results (GC/MS)
7.	Initial calibration results (GC)
8.	Continuing calibration results (GC/MS)
9.	Continuing calibration results (GC)
10.	Internal standard results (GC/MS)
11.	GC analytical sequence
12.	Pesticide cleanup results
13.	Pesticide/PCB identification summary
14.	Method detection limit study results
INORGANIC TABULATED SUMMARY FORMS	
1.	Field sample results
2.	Initial and continuing calibration verification results
3.	Contract required detection limit standard results
4.	Blank results
5.	ICP interference check sample results
6.	Matrix spike and post-digestion spike sample results
7.	Duplicate sample results
8.	Laboratory control sample results
9.	Method of standard additions results
10.	ICP serial dilution results
11.	Instrument detection limits
12.	ICP interelement correction factors
13.	ICP linear ranges
14.	Preparation log
15.	Analysis run log and Furnace AA QC results

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - GENERAL CHECKLIST

DOCUMENTATION	CONTENTS
NARRATIVE	<p>! Laboratory name</p> <p>! Client Request/Contract project number</p> <p>! CLP RAS or other Client sample identification numbers cross-referenced to the laboratory sample identification numbers</p> <p>! Analytical methods and exact procedures performed by the laboratory</p> <p>! Any deviations from the methods</p> <p>! All problems encountered with quality control, samples, shipment, and all analytical problems encountered in processing the samples</p> <p>! Problem resolution must be documented as well as any other factors which may affect the validity of the data</p> <p>! All unusual occurrences encountered during the analysis of the sample set</p> <p>! Explanation of all data flags if not specified in the analytical method</p> <p>! For CLP RAS specific organic data - pH of each water sample submitted for volatiles analysis, list of all instances of manual integrations performed</p> <p>! For CLP RAS specific inorganic data - whether ICP interelement corrections and background corrections were applied</p> <p>! Examples of calculations of both a detected positive result and a detection/quantitation limit reported for each type of sample analysis including all equations, sample volumes, sample weights, dilution factors, percent solids/percent moisture, and other information required to reproduce the laboratory results must be indicated</p> <p>! Signed and dated by the laboratory manager</p> <p>! All telephone communications appended to narrative</p>
LABORATORY LOGBOOK PAGES	<p>Logbook documentation:</p> <p>! Standards preparation logs</p> <p>! QC sample preparation logs</p> <p>! Sample preparation/extraction/digestion logs</p> <p>! Sample analysis run logs</p> <p>! Personal logs</p> <p>! Hand-written project-specific notes</p> <p>Logbook pages contents (where applicable):</p> <p>! Laboratory name</p> <p>! Client sample identification number</p> <p>! Laboratory sample identification number</p> <p>! Dates of preparation and analysis, and initials of preparer/analyst</p> <p>! Source of standards and QC samples</p> <p>! Weights and volumes of samples and standards</p> <p>! Initial and final volumes of sample prepared/purged/extracted/digested</p> <p>! Percent moisture/percent solids</p> <p>! Injection/analysis volumes</p> <p>! Date and time of sample injection</p> <p>! Dilution factors</p>
CHAIN-OF-CUSTODY RECORDS	<p>External Chain-of-Custody Record:</p> <p>! Date of sample receipt</p> <p>! Signature of receiving personnel</p> <p>! Condition of shipping containers and sample bottles upon receipt</p> <p>! Condition of custody seals</p> <p>! Presence/absence of airbills, custody seals, Client custody records, traffic reports, sample tags</p> <p>! Problems or discrepancies with samples received or the documentation on the Chain-of-Custody Record</p> <p>Internal Chain-of-Custody Records:</p> <p>! Laboratory name</p> <p>! Client sample identification number</p> <p>! Laboratory sample identification number</p> <p>! Date of sample transfer and receipt</p> <p>! Signature of personnel transferring/receiving the sample</p> <p>! Purpose of transfer/receipt, location of sample transferred</p>

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
FIELD SAMPLE RESULTS	<p>! Client sample identification (ID) number</p> <p>! Laboratory sample ID number</p> <p>! Target compound names</p> <p>! Tabulated analytical results for identification (numerical quantitation limits) and quantitation (positive hits) with concentration units</p> <p>! Any laboratory qualifier flags - laboratory qualifier flags for each target analyte must be tabulated on a separate form (definitions must be provided for each laboratory qualifier flags).</p> <p>! Laboratory file ID</p> <p>! Sample matrix type</p> <p>! Level of analysis (low, medium)</p> <p>! Percent moisture or percent solids</p> <p>! GC column</p> <p>! Sample weights and/or sample volumes prepared/purged/extracted/analyzed</p> <p>! Initial and final extract and extract clean-up volumes, injection volume</p> <p>! Clean-ups performed</p> <p>! Dilution factor</p> <p>! Measured pH</p> <p>! Dates of sample receipt, extraction, and analysis</p>
SURROGATE RESULTS	<p>! Sample matrix</p> <p>! Level of analysis</p> <p>! GC column</p> <p>! Client sample ID numbers</p> <p>! Surrogate compounds added</p> <p>! Percent recoveries of surrogates</p> <p>! QC limits for all surrogate standards in field samples, QC samples, and blanks</p> <p>! All outliers flagged</p> <p>! Total number of surrogates outside QC limits</p> <p>! Indication of surrogates diluted out</p>
MATRIX SPIKE/MATRIX SPIKE DUPLICATE RESULTS	<p>! Sample matrix</p> <p>! Level of analysis</p> <p>! Client sample ID number</p> <p>! Matrix spike compounds added</p> <p>! True concentrations of the spikes added</p> <p>! Concentrations of the spike compounds observed in the spiked sample</p> <p>! Sample concentration of each spike compound detected in the original unspiked sample for the MS and MSD</p> <p>! Percent recoveries of the spiked compounds in the MS/MSD samples</p> <p>! Relative percent differences of the spiked compounds between the MS and MSD samples</p> <p>! QC limits for all spike compounds - percent recovery and relative percent difference</p> <p>! All outliers flagged</p> <p>In addition to the above, the results for all target compounds in the MS and MSD samples must be tabulated on the summary forms used to tabulate the field sample results.</p>

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
METHOD/LABORATORY BLANK RESULTS	<p>! GC column, instrument ID</p> <p>! Date and time of analysis for the blank itself</p> <p>! Date of extraction</p> <p>! Matrix with which the blank is associated</p> <p>! Level of analysis</p> <p>! Laboratory sample ID number</p> <p>! List of Client field sample ID numbers and MS/MSD samples associated with each blank (separate forms are used for each blank)</p> <p>! Laboratory file IDs of the samples and associated blank</p> <p>! Dates/times of analysis for field samples and MS/MSD samples which are associated with each blank</p> <p>In addition, results for each method and laboratory instrument blank must be included on the tabulated summary forms that are used for the field sample results.</p>
TUNING RESULTS (GC/MS INSTRUMENT PERFORMANCE CHECK)	<p>! Instrument ID, laboratory file ID</p> <p>! Date and time of injection for each tune compound analysis (each tune on a separate form)</p> <p>! Tune compound name</p> <p>! Mass-to-charge ratio (m/e) for each ion</p> <p>! Ion abundance criteria</p> <p>! Percent relative abundances</p> <p>The form must also contain the following tabulated information associated with each tune and in chronological order:</p> <p>! Client sample ID numbers associated with that tune</p> <p>! Laboratory sample ID numbers</p> <p>! Laboratory file IDs</p> <p>! Date and time of analysis for all field samples, MS/MSD samples, blanks, and standards associated with that tune</p> <p>! All outliers flagged</p>
INITIAL CALIBRATION RESULTS (GC/MS)	<p>! Instrument ID, laboratory file IDs</p> <p>! Purge method</p> <p>! Dates and times of standard analyses for that initial calibration</p> <p>! Target compound names</p> <p>! Concentrations of the calibration standards</p> <p>! Relative response factors for each target and surrogate compound at each standard concentration</p> <p>! Mean relative response factors for each target and surrogate compound</p> <p>! Percent relative standard deviations for each target and surrogate compound</p> <p>! QC limits for each initial calibration (each initial calibration on a separate form) - minimum RRF, maximum %RSD values</p> <p>! All outliers flagged</p>

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
INITIAL CALIBRATION RESULTS (GC)	<p>Retention time information:</p> <ul style="list-style-type: none"> ! Instrument ID, GC column ! Dates of analysis ! Concentration of the calibration standards ! Target compound and surrogate compound names ! Retention times for each target and surrogate compound at each standard concentration ! Mean retention times for each target and surrogate compound (if multi-point calibration) ! Retention time windows for each target and surrogate compound (QC limits) <p>Calibration factor information:</p> <ul style="list-style-type: none"> ! Instrument ID, GC column ! Dates of analysis ! Concentrations of the calibration standards ! Target compound and surrogate compound names ! Calibration factors for each target and surrogate compound at each standard concentration ! Mean calibration factor (for multi-point calibration) for each target and surrogate compound ! Percent Relative Standard Deviation for each target and surrogate compound ! QC limits - % RSD ! All outliers flagged <p>Resolution information:</p> <ul style="list-style-type: none"> ! Instrument ID, GC column ! Dates and times of analysis ! Laboratory sample ID ! Names of compounds for which resolution is measured ! Retention times for each of those compounds ! Percent resolution between each pair of compounds ! QC limits - % resolution ! All outliers flagged
CONTINUING CALIBRATION RESULTS (GC/MS)	<ul style="list-style-type: none"> ! Instrument ID, laboratory file ID ! Purge method ! Date and time of continuing calibration analysis ! Date and time of initial calibration analysis associated with that continuing calibration ! Target compound and surrogate compound names ! Mean relative response factors from initial calibration for each target and surrogate compound ! Relative response factors from continuing calibration for each target and surrogate compound (each continuing calibration on a separate form) ! Percent differences for each compound ! QC limits for each target and surrogate compound (each continuing calibration on a separate form) - minimum RRF, maximum % D ! Concentrations of the continuing calibration standards ! All outliers flagged

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
CONTINUING CALIBRATION RESULTS (GC)	<ul style="list-style-type: none"> ! Instrument ID, GC column ! Laboratory sample ID ! Dates and times of continuing calibration standards analysis ! Date of associated initial calibration analysis ! Target compound and surrogate compound names ! Retention time for each target and surrogate compound ! Calculated amount of standard ! Nominal amount of standard ! Relative Percent Difference for each compound ! QC control limits - RPD ! Percent breakdowns for compounds used to measure extent of breakdown (endrin and 4,4'-DDT) and combined breakdown ! QC limits - percent breakdown
INTERNAL STANDARD RESULTS (GC/MS)	<ul style="list-style-type: none"> ! Instrument ID, laboratory file ID ! GC column, purge method ! Date and time of continuing calibration standard analysis ! Client sample identification numbers ! Internal standard compound names ! Retention times and area counts of the quantitation for each internal standard compound in the continuing calibration standard, field samples, MS/MSD samples, and blanks associated with that continuing calibration (separate form for each continuing or initial calibration) ! QC limits - area counts and retention times ! All outliers flagged
GC ANALYTICAL SEQUENCE	<ul style="list-style-type: none"> ! Instrument ID, GC column ! Initial calibration dates ! List of Client sample ID numbers in that analytical sequence (in chronological order) for all standards, field samples, QC samples, and blanks ! Laboratory sample ID numbers ! Dates and times of analyses ! Mean surrogate retention times - from initial calibration ! Retention times of the surrogate compounds ! QC limits of the surrogates - retention times ! All outliers flagged

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
PESTICIDE CLEANUP RESULTS	<p>Florisisl Cartridge Check Results:</p> <ul style="list-style-type: none"> ! GC column ! Florisisl cartridge lot number (each lot on a separate form) ! Date of check solution analysis ! Names of compounds in the Florisisl cartridge check solution ! Amount of spike in the check solution ! Amount of spike recovered in the check solution ! Percent recoveries ! QC limits - percent recoveries ! All outliers flagged ! Client sample ID numbers associated with that Florisisl cartridge ! Laboratory sample ID numbers associated with that Florisisl cartridge ! Dates of sample analysis <p>GPC Calibration Results:</p> <ul style="list-style-type: none"> ! GPC column ! Calibration date of GPC column ! GC columns ! Name of spike compounds added to GPC column ! Amount of spike added ! Amount of spike recovered ! Percent recoveries ! QC limits - % recoveries ! All outliers flagged ! Client sample ID numbers associated with the GPC column calibration ! Laboratory sample ID numbers associated with the GPC column calibration ! Dates of sample analyses
PESTICIDE/PCB IDENTIFICATION SUMMARY	<ul style="list-style-type: none"> ! Instrument ID, GC columns ! Dates of analysis ! Client sample ID number (on a separate form for each sample) ! Laboratory sample ID number ! Target compound name detected ! Retention time of compound on each column ! Retention time windows ! Concentration (mean concentration for multicomponent compounds) ! Percent difference
METHOD DETECTION LIMIT STUDY RESULTS	<ul style="list-style-type: none"> ! Target compound names ! Concentrations of spikes added ! Concentration detected for each MDL spike ! Standard deviation and calculated MDL for each target compound <p>The exact procedure utilized to generate the MDLs must be documented in detail in the narrative. The equation and associated constant values utilized to calculate the MDL for each analysis must be documented. The column, instrument ID, trap composition, and operating conditions must be clearly documented in the raw data.</p>

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - ORGANIC RAW DATA

DOCUMENT	CONTENTS
RAW DATA	<p>The laboratory data package must contain raw data for all field samples, standards, QC samples, matrix spike and matrix spike duplicate samples, and blanks. The exact format and content of the raw data will depend on the particular analysis method requested/contracted. However, all instrument printouts, strip chart recordings, chromatograms, quantitation reports, mass spectra, and other types of raw data generated by the laboratory for a particular project must be provided in the data package.</p> <p>Typical raw data for organic GC/MS analyses includes, but is not limited to the following:</p> <ul style="list-style-type: none"> ! Reconstructed total ion chromatogram for each sample or sample extract, standards, QC samples, and blanks ! Instrument quantitation reports containing the following information: laboratory sample identification number, Client sample identification number, laboratory file ID, date and time of analysis, retention time and/or scan number of quantitation ion with measured area, analyte concentration, copy of area table from data system, GC/MS instrument ID, laboratory file ID, column, trap composition, and operating conditions ! Raw and enhanced mass spectra for all positive target compound results in field samples; daily continuing calibration standard reference spectra for all positive field sample results ! Mass spectra and three library searched best-match mass spectra for all tentatively identified compounds reported ! Instrument normalized mass listing and the mass spectrum for each tune. <p>Typical raw data for organic GC analyses includes, but is not limited to the following:</p> <ul style="list-style-type: none"> ! Chromatograms for field samples, calibration standards, QC samples, and blanks containing the following information: Client sample identification number, volume injected, date and time of injection, GC column identification, GC instrument identification, positively identified compounds must be labeled with the compound names either directly from the peak or on a printout of the retention times ! Chromatograms for both GC columns ! GC integration report or data system printout ! Manual worksheets

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - INORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
FIELD SAMPLE RESULTS	<p>! Sample matrix type</p> <p>! Level of analysis (low, medium)</p> <p>! Percent moisture or percent solids</p> <p>! Date of sample receipt</p> <p>! Client sample identification number</p> <p>! Laboratory sample identification number</p> <p>! Target analyte names</p> <p>! Tabulated analytical results for identification (numerical detection/quantitation limits) and quantitation (positive hits) with concentration units</p> <p>! Any laboratory qualifier flags - laboratory qualifier flags for each target analyte must be tabulated on a separate form (definitions must be provided for each laboratory qualifier flags)</p> <p>! Concentration qualifiers (indicating results less than the CRDL)</p> <p>! Analytical method used for each analyte (usually indicated with a symbol)</p>
INITIAL AND CONTINUING CALIBRATION VERIFICATION RESULTS	<p>! Sources of the initial and continuing calibration verification standards</p> <p>! Target analyte names</p> <p>! True values of the calibration verification standards</p> <p>! Concentrations found for the calibration verification standards</p> <p>! Percent recoveries</p> <p>! QC limits - percent recoveries</p> <p>! Analytical method used for each analyte (usually indicated with a symbol)</p> <p>The order of reporting the initial and continuing calibration verification standards for each analyte must follow the order in which the standards were analyzed.</p>
CONTRACT REQUIRED DETECTION LIMIT STANDARD RESULTS	<p>! Source of the CRDL standards</p> <p>! Target analytes</p> <p>! True values of the CRDL standard for each analyte</p> <p>! Concentrations found for each analyte</p> <p>! Percent recoveries for each analyte</p> <p>! QC limits (if known)</p> <p>The order of reporting the CRDL standard results for each analyte must follow the order in which they were analyzed.</p>
BLANK RESULTS	<p>! Matrix for which the preparation blank is associated</p> <p>! Concentration units for each blank type</p> <p>! Target analyte names</p> <p>! Initial and continuing calibration blank results</p> <p>! Preparation blank results</p> <p>! Concentration qualifiers</p> <p>! Analytical method used for each analyte (usually indicated with a symbol)</p> <p>The order of reporting the initial and continuing calibration blanks and preparation blanks for each analyte must follow the order in which they were analyzed.</p>
ICP INTERFERENCE CHECK SAMPLE RESULTS	<p>! ICP instrument ID number</p> <p>! Source of the ICS solutions</p> <p>! Target ICP analytes</p> <p>! True values of each target ICP analyte in the solution containing interferents only</p> <p>! True values of each target ICP analyte in the solution containing interferents and analytes</p> <p>! Concentrations of target ICP analytes detected in the solution containing interferents only</p> <p>! Concentrations of target ICP analytes detected in the solution containing interferents and analytes</p> <p>! Percent recoveries</p> <p>The order of reporting the interference check sample results for each analyte must follow the order in which they were analyzed.</p>

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - INORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
MATRIX SPIKE AND POST-DIGESTION SPIKE SAMPLE RESULTS	<p>! Sample matrix ! Level of analysis (low, medium) ! Percent solids of the sample ! Client sample identification number ! Target analyte names ! Concentrations of the spikes added to the sample ! Concentrations found in the spiked sample ! Concentration found in the unspiked sample ! Percent recoveries ! QC limits - percent recovery ! All outliers flagged ! Concentration qualifiers ! Analytical method used for each analyte (usually indicated with a symbol)</p> <p>Separate forms are used to report matrix spike results and post-digestion spike results.</p>
DUPLICATE SAMPLE RESULTS	<p>! Sample matrix ! Level of analysis (low, medium) ! Percent solids of the original sample and duplicate sample ! Client sample identification number ! Target analyte names ! Concentration of the original sample result ! Concentration of duplicate sample result ! Relative percent difference ! QC limits ! All outliers flagged ! Concentration qualifiers ! Analytical method used for each analyte (usually indicated with a symbol)</p>
LABORATORY CONTROL SAMPLE RESULTS	<p>! Source of the laboratory control sample ! Matrix of the LCS ! Target analyte names ! True concentrations ! Concentrations found ! Percent recoveries ! QC limits ! Concentration qualifiers</p> <p>Because a laboratory control sample should be digested for each matrix and digestion batch, additional forms must be present as appropriate if more than one LCS for a matrix was analyzed.</p>
METHOD OF STANDARD ADDITIONS RESULTS	<p>! Client sample identification number ! Concentrations of each MSA spike added ! Absorbance detected in each MSA spike as well as the sample itself ! Final concentration ! Correlation coefficient ! All outliers flagged</p> <p>Results for different samples for each analyte must be reported sequentially.</p>
ICP SERIAL DILUTION RESULTS	<p>! Sample matrix ! Level of analysis (low, medium) ! ICP instrument ID ! Client sample identification number ! Target analyte names ! Concentrations of the undiluted sample result ! Concentrations of the diluted sample result ! Percent difference ! QC limits - percent difference ! All outliers flagged ! Concentration qualifiers ! Analytical method used for each analyte (usually indicated with a symbol)</p>

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - INORGANIC TABULATED SUMMARY FORMS

DOCUMENT	CONTENTS
INSTRUMENT DETECTION LIMITS	<ul style="list-style-type: none"> ! Instrument ID numbers used for the IDL determination ! Date the IDLs were determined ! Wavelength and background used for each analyte ! Target analyte names ! Type of background correction used where applicable ! Instrument detection limits ! Contract required detection limits ! Concentration units ! Analytical method used for each analyte (usually indicated with a symbol)
ICP INTERELEMENT CORRECTION FACTORS	<ul style="list-style-type: none"> ! Wavelength for each analyte used for the determination ! ICP instrument ID number ! Date of interelement correction factor determination ! Target ICP analyte names ! Interfering analytes with which the interelement correction factors were determined ! Interelement correction factors for each analyte
ICP LINEAR RANGES	<ul style="list-style-type: none"> ! ICP instrument ID number ! Date of the linear range determination ! Integration time for each analyte ! Target ICP analytes ! Concentration of the upper limit of the linear range for each analyte
PREPARATION LOG	<ul style="list-style-type: none"> ! Analytical method (each analytical method on a separate form) ! Client sample ID number of all field samples, QC samples, standards, and blanks digested/distilled ! Sample preparation date ! Sample weight ! Sample volume
ANALYSIS RUN LOG	<ul style="list-style-type: none"> ! Instrument ID ! Analytical method ! Start and end dates of the analytical sequence ! Client sample ID numbers in chronological order ! Dilution factors ! Time of analysis for each analytical sample and standard ! Analytes for which the run sequence pertains <p>Furnace AA QC analyses results are also typically provided on this form - see below.</p>
FURNACE AA QC RESULTS	<p>In addition to the information required on the Analysis Run Log (see above):</p> <ul style="list-style-type: none"> ! Percent relative standard deviation of duplicate injections (outliers usually indicated with flags on the tabulated field sample results summary form, Form 1) ! Percent recoveries of post-digestion spikes <p>The above information must be reported on separate forms for each furnace AA analyte.</p>

LABORATORY DATA PACKAGE COMPLETENESS

CONTENTS - INORGANIC RAW DATA

DOCUMENT	CONTENTS
RAW DATA	<p>The laboratory data package must contain raw data for all field samples, standards, QC samples, matrix spike and duplicate samples, and blanks. The exact format and content of the raw data will depend on the particular analysis method requested/contracted. However, for each reported value for a particular project, the laboratory must include all raw data used to obtain that reported value.</p> <p>Typical raw data for inorganic analyses include, but is not limited to the following:</p> <ul style="list-style-type: none">! Instrument printouts, strip chart recordings, etc., for all field samples, QC samples, standards, and blanks containing the following information: laboratory sample identification number, Client sample ID number, date and time of analysis, absorbance/emission values, analyte concentration, instrument ID, lab file ID, and instrument operating conditions.! Standard curve raw data, plotted standard curves, linear regression equations, and correlation coefficients.

ATTACHMENT VI

REGION I CSF COMPLETENESS EVIDENCE AUDIT PROGRAM

JULY 3, 1991

(See Attachment C of the Functional Guidelines)